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THE CARBONIZATION OF MISSOURI CANNEL COALS

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SCHOOL OF MINES
AND METALLURGY
UNIVERSITY OF MISSOURI



THE CARBONIZATION OF
MISSOURI CANNEL COALS

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ROLLA, MO.

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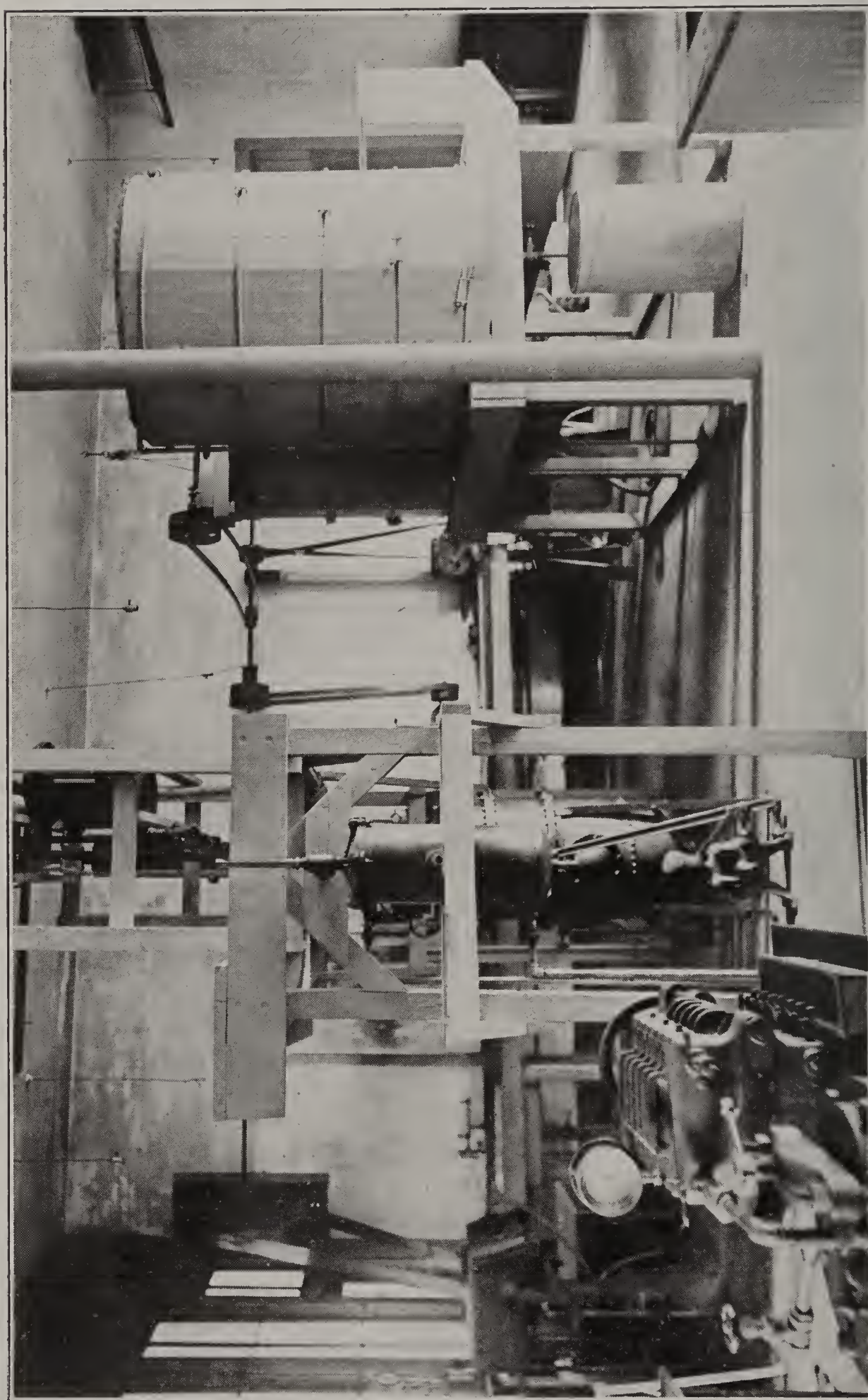
H. L. Wheeler

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MATERIALS TESTING LABORATORY.

BULLETIN

AUGUST, 1919

School of Mines and Metallurgy

UNIVERSITY OF MISSOURI

TECHNICAL SERIES

Vol. V.

AUGUST, 1919.

No. 1

THE CARBONIZATION OF MISSOURI CANNEL COALS

Introduction

This bulletin discusses investigations which were undertaken in continuation of the work described in "Studies in the production of oils and tars from bituminous materials," by J. C. Ingram¹. The present report deals primarily with the cannel coals of Missouri.

The importance of a thorough examination of cannel coals is due to the fact that many of them have a high percentage of volatile matter. Upon destructive distillation these coals yield a product which is more of the nature of an oil than of a tar such as is obtained from ordinary coal, and this fact has suggested the idea that these oils might have a commercial value much different from bituminous coals.

The data presented should be considered as a preliminary study of the general nature of the distillation products, rather than as a detailed investigation with a wide range of conditions in carbonization.

¹Missouri School of Mines and Metallurgy, Bulletin, Technical Series, Vol. 3, No. 4; May, 1917.

General Considerations

The time², temperature, pressure, shape and size of the retort³ have a very marked influence upon the products obtained by the carbonization of any coal. The products of carbonization are in three phases,—solid, liquid and gaseous⁴, and the constituents and yield of these three phases may be widely varied by comparatively slight changes in the factors mentioned above. The influence of the physical conditions under which a coal is carbonized can not be over-emphasized.

The lapse of time between the mining of a coal and the subsequent distillation has an influence upon the data obtained. Unfortunately, this lapse of time, owing to transportation difficulties, is not uniform. Care in selecting a representative sample of a coal is also necessary. Uniform crushing of the sample, in so far as hardness and cleavage will permit, is essential in order to secure comparative data. The weight of coal used in each test must be governed by the facilities for proper coking and for the necessary handling of the oils and gases given off.

The size, shape and cooling facilities of the condensers will affect the products, both oils and gases, as secondary reactions take place at this stage in the process. If the gases are required to pass over a heated path, it is evident that the cracking of the oils will be greater. The longer that the hot gaseous products remain in this state the greater this cracking will be.

The pressure under which the coal is distilled also affects the products, since a higher pressure would require a higher temperature for volatilization of the constituents; and this higher temperature would tend to cause a still greater cracking of the oils. In the condensing of the gases, also, the increase or decrease of the pressure would bring about more or less polymerization, while the relative amounts of paraffins and olefines would for the same reason be altered.

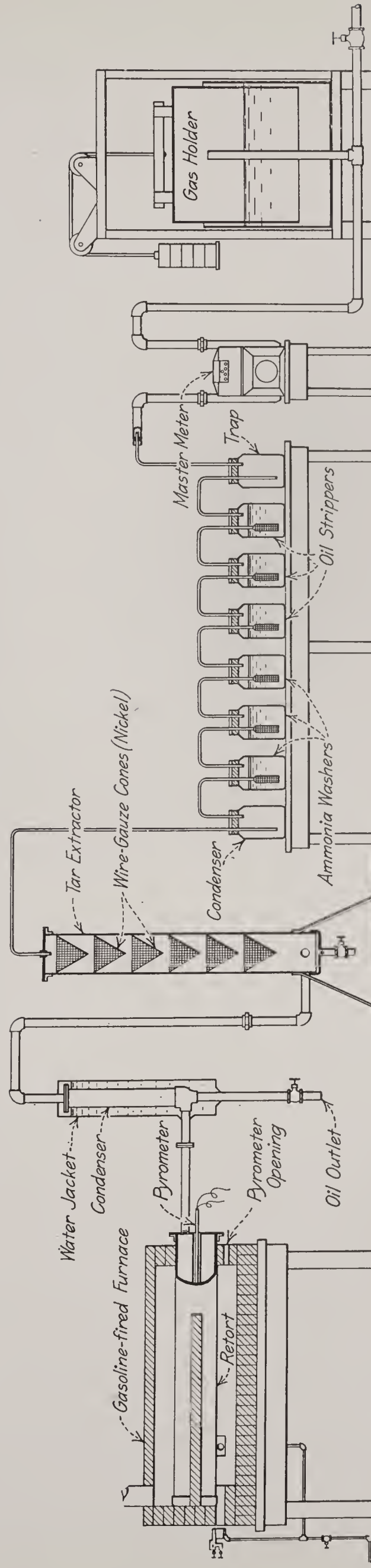
In heating the retort, the volatile products from the coal adjacent to the walls⁵ are driven to the interior and condensed, then redistilled later when the temperature of this part of the retort reaches the proper stage. These volatile products would evidently act as a solvent for the other volatile products and alter the decomposition point of the coal in the interior.

²Jayne, H. W., Coal tar industries in the United States; Fifth International Congress of Applied Science, Vol. 2, p. 721, 1906.

³Perry, R. P., Tar distillation in the United States: Eighth International Congress of Applied Science, Vol. 19, p. 233, 1912.

⁴Wagner, F. H., Coal gas residuals, p. 7, 1914.

⁵Traer, G. W., Low temperature distillation of Illinois and Indiana coals: Am. Inst. Min. Eng., Bull. 141, pp. 1463-1470, Sept., 1918.



APPARATUS FOR CARBONIZATION OF COALS AND THE RECOVERY OF BY-PRODUCTS.

Description of Apparatus

The apparatus used for this work was of the same general type as that employed by J. C. Ingram in work previously carried out at the Missouri School of Mines and Metallurgy. However, certain modifications were made.

Furnace.—The furnace was the same in principle but was lengthened to accommodate a larger retort. The method of heating was the same, namely, by three gasoline burners.

Retort.—The retort was made from a six-inch steel pipe, five feet long, and had a capacity of about one cubic foot. It was closed at the rear with a cast iron cap; the front end was fitted with a half flange, and this was capped with a plate having an outlet to the first condenser. This plate was also fitted with a three-quarter inch capped iron tube extending back thirty-six inches through the center of the retort. This was used for inserting the thermo-couple in order to secure, as nearly as possible, the temperature of the interior of the retort. The furnace was so constructed that the flames from the burners played along the under side of the retort towards the front end, and then back over the top to rear of the furnace. From several readings, it was found that the temperature of the outside of the retort was approximately one hundred degrees centigrade higher than that of the inside. The temperature of the inside was recorded in the data.

Condensers.—The first condenser was the same as that previously used and was cooled by running water during the entire distillation. The second condenser consisted of four and one-half feet of five-inch steel pipe with six neatly-fitting nickel wire gauze cones as shown in the diagram. The third condenser was a five-liter bottle. It was necessary to insert this in order to give the tars an opportunity completely to settle out of the gases. The connection between the first and second condensers was a two-inch iron pipe, and a half-inch glass tube joined the second and third.

Ammonia washers.—Four ten-liter glass acid bottles served as ammonia washers. Five liters of water were placed in each washer and the gas bubbles were scattered by placing a nickel wire gauze cap over each inlet. This proved very efficient, as rarely was there found to be any ammonia in the third washer. The first washer was fitted with a manometer for determining the pressure at any time.

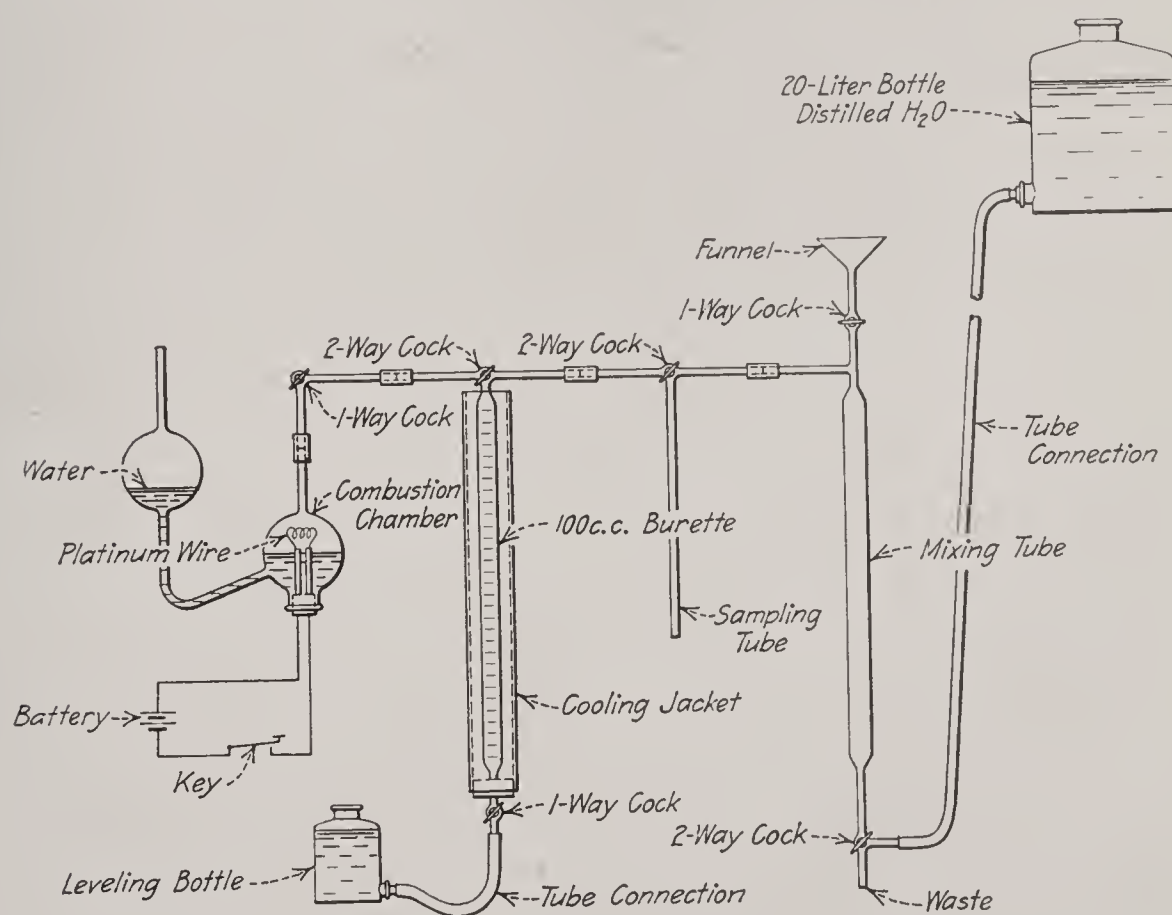
Toluol, benzol, and xylol absorbers.—Three five-liter bottles for removing these constituents were arranged similar to the ammonia washers and the gas caused to scatter in the same manner. The oil used in the washers was kindly furnished by the

Laclede Gas Light Co. of St. Louis, and was of the same kind as that used for this purpose in their plants. The connections between these absorbers and the ammonia washers consisted of half-inch glass tubing.

The toluol, benzol, and xylol absorbers were joined to a master meter by means of a two-inch iron pipe, and the master meter was in turn joined in the same manner to the reservoir tank.

Gas tank.—The reservoir tank was constructed of galvanized iron and had a capacity of approximately fifty cubic feet. In order to secure a general sample of the gases for the purpose of determining the B. t. u. value and also for analysis, a suitable amount of coal was placed in the retort to enable the tank to hold the complete output of gases.

The pressure required to force the gases through the entire apparatus was two centimeters of mercury. This was a greater pressure than was desired, but no way was found to lessen it and at the same time completely wash the gas. The pressure was constant for all runs.



APPARATUS FOR ANALYZING GAS.

Gas apparatus.—The gas samples were analyzed by means of a modified Elliot apparatus. The modifications were necessary in order to secure quick and satisfactory results with this particular gas, which ran very high in hydrogen and methane. The standard Elliot apparatus consists of three interconnected, 100 c. c. burettes. The first is not graduated and is used as a reaction chamber for treating the gas with the several chemicals used.

The second is graduated and is used for measuring the volume of the gas before and after treatment in the first burette. The third burette is similar to the second except that it has a spark gap near the top and is used for the explosion of the hydrogen and methane. The system is connected to a supply of distilled water and a leveling bottle is fitted to the bottom of each burette.

The modified apparatus retained the first and second burettes but substituted a slow combustion pipette for the third. It was found that the high percentage of methane and hydrogen rendered the explosion method inaccurate as well as dangerous for the operator. The combustions in the pipette were made over water at atmospheric pressure with ignition by means of an electrically heated platinum wire. The only other modification consisted in connecting the bottom of the first burette with a twenty-liter bottle containing distilled water and placed about four feet overhead. Connection was made by means of a two-way cock, one branch leading to the water supply and the other to the waste. This change did away with the necessity for leveling bottles on the first and third burettes and made the operation of the apparatus much simpler. The whole apparatus was mounted on a rigid frame.

The manipulation consisted briefly in drawing 100 c. c. of gas into the second burette from the gas holder, then transferring this charge to the reaction burette under slightly reduced pressure, and sucking in a few drops of the desired chemical through a capillary tube at the top of the burette. As soon as the reaction was complete the gas was returned to the second burette to be measured, and the first burette was flushed out with distilled water from the two-way valve. This operation was repeated with each reagent used. The combustion was made by drawing 15 c. c. of gas into the combustion pipette, turning on the current through the platinum wire, and then forcing in 100 c. c. of air from the second burette. The reduction of volume was also measured in this burette. Oxygen was used for a portion of the combustion, thus making it possible to use a full pipette of the gas (100 c. c.) instead of the 15 c. c. used with the air.

Analyses made with this apparatus were found to agree closely with those made with the standard Hempel apparatus. It was easily possible to make a complete determination in thirty minutes, and the apparatus gave practically no trouble during the course of the work.

Examination of the Oils

The tars or oils secured from the different runs of the same coal did not, as far as could be determined, differ much in their properties. A general sample of the runs was taken for the examination of each coal.

In the examination of these oils, the methods used by the Barrett Manufacturing Company were followed. These methods, as Mr. Church states, are not "for the scientific examination of, or into, the products of coal tar." The primary object was to secure concordant results in different laboratories.

At the suggestion of Mr. J. M. Weiss, head of the Research Department of the Barrett Manufacturing Company, a sample of the crude Bunceton oil was sent to the New York laboratory for examination. Mr. Weiss, commenting upon it, writes:

"The tar acid content is low, and not what we would expect from low temperature tars, as in some cases we have seen these run as high as 25 to 30 per cent of acid bodies. Although the oil contains considerable material boiling below 200 degrees, yet in view of the 40 per cent paraffin content, we do not believe that this could be utilized for the production of commercial aromatic hydrocarbons."

The cannel coal oil when freshly fractionated was of a light straw color for the first fraction, and a darker color for each succeeding fraction. Upon standing, all of these fractions became darker in color. In a light room, but not in the sunlight, all the samples became black in color after three months and left a tarry sediment in the bottom of the flask. Another set of samples exposed to more light darkened much faster. When the fractions were kept in the dark, the blackening was very slow; but upon standing, there was a gradual change, showing some decomposition even in the absence of light.

Several of the fractions were redistilled. It was found that some of the oil began to come over at a temperature lower than where the cut was made, and resembled in physical properties the oils in the lower-boiling fractions. At the maximum temperature of the cut, there would always upon redistillation be an oil remaining, darker in color, which resembled the next higher fractions. This would indicate that the higher hydrocarbons were continually being broken up into the simpler compounds on the one hand, while at the same time some condensations were taking place. This was probably due to the unsaturated compounds, as the bromine number of the oils of the same specific gravities showed a decrease upon these redistillations.

The two lower fractions of the cannel coal oils were tested for benzene according to Mr. Church's method⁶, but gave only very slight traces. It may be possible to crack these oils and obtain benzene and toluene. It is probable that they would lend themselves easily to a process of this kind on account of their instability when subjected to repeated fractional distillations.

In the fractions of these oils there is a mixture of compounds⁷, paraffins, olefines, and varying amounts of aromatic hy-

⁶Church, S. R., *Methods for testing coal tar and refined tars, oils and pitches*: Jour. Ind. & Eng. Chem., Vol. 3, pp. 227-233, 1911; also Vol. 5, pp. 195-197, 1913.

⁷Jones, D. T., and Wheeler, R. V., *The composition of coal*: Jour. Chem. Soc., Vol. 105, pp. 140-151, 1914.

drocarbon derivatives. A separation by fractional distillation would be impossible, owing to the breaking down of some and the condensation of others. An attempt was made to nitrate some of the fractions and then crystallize them. Some fairly good crystals were obtained from some of the lower fractions but any further attempts always led to a tarry mass with which it was impossible to proceed further. With the higher fractions it was found more difficult to get anything as definite as with the lower fractions.

Parr and Olin⁸ raised the question whether such tars could not be used as drying bodies or paint vehicles. A quantitative measurement of the capacity for absorbing bromine⁹ was run on the different fractions where possible and recorded in the data. Great difficulty was encountered in getting a good end point. The bromine number was calculated as grams absorbed by one hundred grams oil. The best results were obtained by using carbon tetrachloride as a solvent for the bromine. For comparative results, it was necessary to run these bromine numbers under the same conditions of temperature and time. After titrating the iodine displaced against sodium thiosulphate and letting the solution stand for a few minutes, the oil became dark and in some of the higher fractions a tarry substance separated similar to that which was formed upon standing exposed to the light.

In the determination of the tar acids and tar bases, definite readings were easily obtained for the lower fractions but some of the higher boiling fractions required considerable time for the separations. The same general tendency was noticeable in getting the unsulphonated residue readings. The fractions obtained by fractionation were much more easily read than were those of the crude tars. An oily heavy tar persists in adhering to the surface of the pipette, making reading impossible.

Description of coals used

Six different samples of coal were examined. As soon as the samples were received, they were crushed into one-half to one-fourth inch lumps and placed in closed containers until ready to be used.

Bunceton coal.—This sample was obtained from the Hubbard Cannel Coal Mine near Bunceton, Cooper County, Missouri, sec. 29, T. 47 N., R. 16 W. This mine has been worked eight years. The extent of the pocket has not been definitely determined, but it is estimated to be 50 feet thick with a width of 300 feet and a length of 500 feet. The depth from the surface is about 55 feet.

⁸Parr, S. W., and Olin, H. L., Univ. of Illinois Eng. Exper. Sta., Bull. 60, p. 18, 1912.

⁹Allen, A. H., Commercial organic analysis, Vol. 2, p. 26, 1909.

The coal breaks out in large blocks and splinters upon crushing. It fuses when heated and upon ignition burns with a smoky flame. The following is an analysis of the coal, coke and ash:

Analysis of Bunceton coal.

Volatile matter	51.42	
Ash	8.16	
Moisture	0.82	
Fixed carbon	39.60	
	<hr/>	
	100.00	
Specific gravity, apparent	1.290	
B. t. u.	14,208	
Ammonium sulphate, per ton	12.41	pounds
Sulphur	3.19	per cent

Analysis of ash from Bunceton coal.

Insoluble residue	55.50
Fe ₂ O ₃	21.60
Al ₂ O ₃	19.30
CaO	1.29
Sulphur	1.85
	<hr/>
	99.54

Analysis of coke from Bunceton coal.

Sulphur	2.19	per cent
B. t. u.	11,714	

This coal gave a very good coke. It was hard and the entire charge fused together. The porousness was about the same as that of ordinary coke.

Ouachita coal.—The sample of Ouachita coal was taken from the Ouachita mine, Morgan County, Missouri, SW.¼ SW.¼ sec. 22, T. 43 N., R. 18W., about seven miles northwest of Versailles, Missouri.

There are several pockets of the coal in this region and some of these have been worked for local consumption for several years. The mine at present is being put into operation by H. H. Hannenkratt and a branch switch repaired to the mine. The sample secured can hardly be considered typical of the deposit, as it was impossible to get into the deposit far enough to get away from effect of exposure to the air. The sample was jet black and gave the characteristic cannel coal splintering upon crushing. It burns with a smoky flame and gives a high per cent ash, but lower per cent of volatile matter than some other cannel coals. The approximate dimensions of the pocket examined are, thickness 45 feet, width 200 feet, length undetermined. The deposit is about 15 feet below the surface of the ground. The analysis is as follows:

Analysis of Ouachita coal.

Volatile matter	25.49	
Ash	55.06	
Moisture	4.90	
Fixed carbon	14.55	
	<hr/>	
	100.00	
Specific gravity, apparent	1.641	
B. t. u.	5,950	
Ammonium sulphate, per ton	5.20	pounds
Sulphur	0.89	per cent

Analysis of ash from Ouachita coal.

Insoluble residue	94.00
Fe ₂ O ₃ and Al ₂ O ₃	3.50
CaO	0.35
Sulphur	0.30
	<hr/>
	98.15

Analysis of coke from Ouachita coal.

Sulphur	1.07	per cent
B. t. u.	6,415	

This coal gave a retort residue which did not in the least clinker. Thus it would have no value as a coke for fuel.

Moniteau coal.—This deposit is located in Moniteau County, Missouri, NW. $\frac{1}{4}$ sec. 16, T. 43 N., R. 16W., about twelve miles northwest of Versailles, Morgan County. The deposit is about 20 feet below the surface of the ground; it has a thickness of about 42 feet, a width of 400 feet and length undetermined. The mine has been worked for local consumption for fifty years and is being worked at present. The coal crushes into characteristic cannel coal splinters, runs comparatively low in ash and high in volatile matter. The following is the analysis of the coal, coke and ash:

Analysis of Moniteau coal.

Volatile matter	48.36	
Ash	9.10	
Moisture	2.07	
Fixed carbon	40.47	
	<hr/>	
	100.00	
Specific gravity, apparent	1.118	
B. t. u.	14,318	
Ammonium sulphate, per ton.	10.10	pounds
Sulphur	2.12	per cent

Analysis of ash from Moniteau coal.

Insoluble residue	90.90
Al ₂ O ₃ and Fe ₂ O ₃	7.00
CaO	2.00
Sulphur	0.80
	<hr/>
	100.00

Analysis of coke from Moniteau coal.

Sulphur	1.50	per cent
B. t. u.	11,242	

This coal gave a light inferior grade of coke which did not clinker to any very great extent.

Stover coal.—This deposit is located in Morgan County, Missouri, SE.¼ NE.¼ sec. 6, T. 41 N., R. 16W., near Barnett, Missouri. The mine has been worked intemittently for the last fifty years. The depth of the deposit from the surface is 10 to 20 feet and the bed is over 70 feet in thickness; the width is unknown but there is a side drift about 200 feet from the entrance of the tunnel, which measures 120 feet in solid coal. There are also two or three smaller drifts. The length of the drift is unknown but at present there is a tunnel approximately 400 feet long in solid bituminous formation. The coal from this mine resembles bituminous coal in appearance; it is jet black and in breaking up shows some of the characteristics of cannel coals. It runs exceptionally low in ash and high in volatile matter. The following is an analysis of the coal, coke and ash:

Analysis of Stover coal.

Volatile matter	49.10	
Ash	2.30	
Moisture	7.20	
Fixed carbon	41.40	
	<hr/>	
	100.00	
Specific gravity, apparent	1.381	
B. t. u.	14,257	
Ammonium sulphate, per ton.....	10.12	pounds
Sulphur	4.80	per cent

Analysis of ash from Stover coal.

Insoluble residue	92.00
Fe ₂ O ₃	5.30
Al ₂ O ₃	2.10
CaO	0.30
Sulphur	2.10
	<hr/>
	101.80

Analysis of coke from Stover coal.

Sulphur	3.05	per cent
B. t. u.	13,574	

This coal gave a good coke but runs high in sulphur.

Maryville coal.—This coal came from an Illinois mine near Maryville and is typical of the Illinois bituminous coals. The sample was selected for comparison with the Missouri cannel coals used in the retorts.

Analysis of Maryville coal.

Volatile matter	40.90	
Ash	9.00	
Moisture	8.70	
Fixed carbon	41.40	
	<hr/>	
	100.00	
Specific gravity, apparent	1.294	
B. t. u.	12,218	
Ammonium sulphate, per ton	30.20	pounds
Sulphur	3.70	per cent

Analysis of ash from Maryville coal.

Silica	53.66
Fe ₂ O ₃	18.18
Al ₂ O ₃	24.18
CaO	3.33
MgO	0.80
Sulphur	0.50
	<hr/>
	100.65

Analysis of coke from Maryville coal.

Sulphur	2.74	per cent
B. t. u.	11,954	

This coal gave a harder coke than did any of the cannel coals examined, but to get a good coke a higher temperature than that obtained in our retorts would be required, and also a longer time for heating would be necessary.

Wyoming coal.—A sample of this coal was at hand and it was run. This coal is a very poor quality, being very soft and brittle. The sample was taken from the Brook mine, Campbell County, Wyoming, T. 50, R. 72. The deposit is about 22 feet thick and of unknown extent. The deposit has been worked 5 years for local use.

Analysis of Wyoming coal.

Volatile matter	65.40	
Ash	2.42	
Moisture	20.20	
Fixed carbon	11.98	
	<hr/>	
	100.00	
Specific gravity, apparent	1.440	
B. t. u.	10,980	
Ammonium sulphate, per ton	15.80	pounds
Sulphur	.043	per cent

Analysis of ash from Wyoming coal.

Insoluble residue	22.49
Fe ₂ O ₃ and Al ₂ O ₃	27.39
CaO	43.56
Sulphur	5.56
Alkalies	1.00
	<hr/>
	100.00

Analysis of coke from Wyoming coal.

Sulphur	0.65	per cent
B. t. u.	12,325	

The retort residue showed no signs of clinkering. It gave a powdered residue, almost a dust, but a very good B. t. u., and has a low sulphur content. It would have no commercial value as a fuel on account of its physical state.

Data from the distillation of the coals examined.

NOTE: Readings for all runs were taken every half hour and the numbers above each temperature indicate each consecutive half hour.

BUNCETON COAL

Destructive distillation of 20 lb. of Bunceton coal.

	1	2	3	4	5	6	7	8	9	10	Totals
Time, half hours	350	375	425	495	580	660	782	860	980	980	
Temperature, degrees C.	40	25	345	640	100	25					1175
Oil, first condenser, c. c.	5	46	93	51	28	18					241
Water, first condenser, c. c.	.8214	.8387	.8596	.8772	.8811	.8965					
Oil, first condenser, sp. gr.	25	10	10	155	250	200	100				750
Oil, second condenser, c. c.											
Water, second condenser, c. c.											
Oil, second condenser, sp. gr.	.8769			.9106	.9362		.9368	.9527			
Oil, third condenser, c. c.	100	c. c. taken as a drain at end of run									100
Oil, third condenser, sp. gr.	.9847										
Gas, cubic feet	1	1	10	34	38		16	3			103

A ton of coal was found to give, according to this run 53.50 gal. of oil; 5.09 gal. of water; 1080 lb. coke; and 10,264 cu. ft. of gas.

Destructive distillation of 24 lb. of Bunceton coal.

	1	2	3	4	5	6	7	8	9	10	11	Totals
Time, half hours	300	460	530	550	575	625	663	683	760	760	760	
Temperature, degrees C.	10	490	800	300	100	10						1710
Oil, first condenser, c. c.	20	191	65	42								318
Water, first condenser, c. c.		5	200	250	10							365
Oil, second condenser, c. c.												
Water, second condenser, c. c.												
Oil, third condenser, c. c.	120	c. c. taken as a drain at end of run										120
Gas, cubic feet	5	8	18	27	20	8	2	3	2	2	3	98

The specific gravity for the first, second and third condensers was the same as that given for the first run.

A ton of coal was found to give, according to this run, 48.32 gal. of oil; 7.00 gal. of water, 1080 lb. of coke; and 8208 cu. ft. of gas.

Analysis of a general sample of the gas.

N ₂	12.7
H ₂ S and SO ₂	3.4
CO ₂	1.5
Illuminants	15.6
O ₂	2.5
CO	0.8
CH ₄	32.5
H ₂	31.2
Total	100.2

The general sample gave a B. t. u. value of 997.

Analysis of the gas given off at end of each half hour during the distillation of the coal.

Time, half hours	1	2	3	4	5	6	7	8	9	10	11	12	13
N ₂ ^a	-----	-----	-----	15.4	-----	4.9	-----	7.7	-----	3.7	-----	4.3	1.6
H ₂ S and SO ₂	-----	-----	-----	5.5	-----	1.7	-----	1.0	-----	0.5	-----	0.2	0.2
CO ₂	-----	-----	-----	7.2	-----	12.1	-----	13.5	-----	17.2	-----	2.3	1.3
Illuminants	-----	-----	-----	10.0	-----	4.2	-----	1.1	-----	1.5	-----	1.1	1.2
O ₂	-----	-----	-----	2.3	-----	1.3	-----	1.4	-----	0.9	-----	-----	0.3
CO	-----	-----	-----	1.3	-----	1.1	-----	1.6	-----	1.1	-----	1.8	2.4
CH ₄	-----	-----	-----	23.2	-----	42.1	-----	17.5	-----	22.4	-----	20.1	17.0
H ₂	-----	-----	-----	35.1	-----	32.6	-----	56.2(?)	-----	52.7	-----	70.2	76.0
Totals	-----	-----	-----	100.0	-----	100.0	-----	100.0	-----	100.0	-----	100.0	100.0

^aNitrogen obtained by difference.

Analysis of the fractions from the fractionation of the general oils.

Temperature, degrees C.	70-125	125-170	170-200	200-210	210-235	235-270	270-305	305-320	320-355	355-400
Specific gravity7711	.8005	.8350	.8541	.8768	.8925	90.91	.9265	.9460	.9489
Fixed carbon, per cent04	.05	.05	.05	.07	.09	.17	.27	.29	.74
Volatile matter, per cent	99.96	99.94	99.94	99.94	99.92	99.90	99.82	99.72	99.70	99.25
Ash, per cent00	.01	.01	.01	.01	.01	.01	.01	.01	.01
Limpid point	below	C. for first six fractions								
Flashing point	50°F.	100°F.	120°F.	150°F.	180°F.	210°F.	250°F.	320°F.	320°F.	30°F.
Burning point	75°F.	140°F.	160°F.	210°F.	265°F.	280°F.	320°F.	-----	-----	-----
Tar acid, per cent.	1.7	2.0	10.0	11.0	10.0	8.0	7.0	6.0	8.0	9.5
Tar base, per cent	0.0	0.5	0.0	0.0	1.0	1.0	2.0	3.0	2.5	2.5
Viscosity at 60 degrees C.	1.04	1.09	1.10	1.11	1.12	1.15	1.26	1.42	1.66	2.16
Per cent of amount	2.4	11.4	6.4	3.6	7.2	13.4	9.8	6.0	8.4	14.6
Bromine number	31.48	33.89	37.73	37.28	-----	38.89	33.53	33.16	34.75	35.11
Unsulphonated residue, per cent	55.70	66.66	75.00	70.10	64.30	50.00	50.00	45.00	40.00	33.10

Analysis of a general sample of the oil.

Specific gravity9152
Water, per cent	6.60
Flashing point, degrees F.	110.00
Burning point, degrees F.	200.00
Viscosity, 60 degrees C.	1.16
Tar acid, per cent	6.80
Tar base, per cent	1.00
Volatile matter, per cent	91.81
Ash, per cent16
Fixed carbon, per cent	2.02
Free carbon, per cent	7.04
Napthalene, per cent	none
Limpid point, degrees C.	1.5
Unsulphonated residue, per cent	15.00

Comparison of Bunceton coal tar with ordinary tar in fractionating^a

Constituent	Bunceton tar, per cent	Ordinary tar, per cent
Water	6.6	15.0
Light oil	14.0	1.4
Middle oil	17.2	4.2
Heavy oil	13.4	24.0
Anthracene oil	38.8	.2
Pitch	10.0	55.0

^aCohen, Theoretical organic chemistry, p. 381.

OUACHITA COAL.

Destructive distillation of 25 lb. of Ouachita coal.

Time, half hours	1	2	3	4	5	6	7	8	9	Totals
Temperature, degrees C.	-----	315	425	475	525	550	600	635	750	1140
Oil, first condenser, c. c.	-----	-----	10	70	620	290	120	20	10	1140
Water, first condenser, c. c.	-----	-----	360	350	350	50	30	-----	-----	-----
Oil, first condenser, sp. gr.	-----	-----	.8672	.8758	.8845	.8988	.9656	.9791	.9761	5
Oil, second condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	-----	5	-----
Water, second condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
Oil, third condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	-----	-----	145
Oil, third condenser, sp. gr.	-----	1.0274 for the drain.	1	1	7	19	16	1	-----	45
Gas, cubic feet	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
A ton of coal was found to give, according to this run, 27.79 gal. of oil; 24.09 gal of water; 1520 lb. coke; and 3600 cu. ft. gas.										

Destructive distillation of 25 lb. of Ouachita coal.

Time, half hours	1	2	3	4	5	6	7	8	9	Totals
Temperature, degrees C.	-----	340	390	440	550	650	700	825	850	1019
Oil, first condenser, c. c.	-----	24	100	325	395	100	40	10	25	1171
Water, first condenser, c. c.	-----	25	600	405	125	10	-----	-----	6	-----
Oil, second condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	20	-----	20
Water, second condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	35	-----	35
Oil, third condenser, c. c.	-----	-----	-----	-----	-----	-----	-----	-----	-----	150
Gas, cubic feet	-----	-----	1	7	15	13	10	3	-----	49

The specific gravity for the first and second condensers was the same as that found for the first run.

A ton of coal was found to give, according to this run, 25.13 gal. of oil; 24.49 gal. of water; 1600 lb. of coke; and 3920 cu. ft. of gas.

Analysis of a general sample of the gas.

N ₂	11.9
H ₂ S and SO ₂	1.8
CO ₂	1.9
Illuminants	5.7
O ₂	2.6
CO	3.7
CH ₄	38.2
H ₂	34.3
Total	100.1

The general sample gave a B. t. u. value of 834.

Analysis of the gas given off at the end of each half hour during the distillation of the coal.

Time, half hours	1	2	3	4	5	6	7
N ₂	-----	-----	23.4	4.0	3.2	1.9	-----
H ₂ S and SO ₂	-----	-----	-----	0.9	2.3	1.7	1.7
CO ₂	-----	-----	-----	3.7	3.1	2.4	2.8
Illuminants.	-----	-----	2.0	5.4	6.2	9.5	8.3
O ₂	-----	-----	2.2	1.2	1.4	1.0	1.0
CO.	-----	-----	5.0	1.3	2.1	1.9	1.8
CH ₄	-----	-----	15.3	34.0	35.2	34.8	34.6
H ₂	-----	-----	52.2	49.0	48.3	48.7	49.1
Totals	-----	-----	101.1	99.5	101.8	101.9	99.3

Analysis of the fractions from the fractionation of the general oils.

Temperature, degrees C.	70-125	125-170	170-200	200-235	235-270	270-305	305-320	320-355	355-400
Specific gravity7644	.7806	.8109	.8346	.8571	.8777	.8964	.9258	.9357
Fixed carbon, per cent05	.05	.05	.19	.19	.19	.19	.51	.66
Volatile matter, per cent	99.95	99.93	99.93	99.81	99.81	99.81	99.81	99.47	99.36
Ash, per cent00	.02	.02	.02	.02	.02	.02	.02	.02
Limpid point	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.	below -10°C. for the first five fractions.
Flashing point	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.	130°F. for first four fractions.
Burning point	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.	140°F. for first four fractions.
Tar acid, per cent	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions	7 for first four fractions
Tar base, per cent	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions	0 for first four fractions
Viscosity at 60 degrees C.	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions	1.07 for first four fractions
Per cent of amount	2.7	5.6	6.7	16.2	12.6	9.7	6.0	11.7	13.0
Bromine number	58.11	53.74	53.88	52.16	51.47	49.81	47.44	(not enough)	-----
Unsulphonated residue, per cent	-----	-----	-----	-----	-----	-----	4.25	-----	-----

Analysis of a general sample of the oil.

Specific gravity9081
Water, per cent	11.30
Flashing point, degrees F.	90
Burning point, degrees F.	105
Viscosity, 60 degrees C.	1.48
Tar acid, per cent	8.33
Tar base, per cent	0.00
Volatile matter, per cent	99.30
Ash, per cent03
Fixed carbon, per cent	1.67
Free carbon, per cent87
Napthalene, per cent	0.00
Limpid point, degrees C.	4
Unsulphonated residue, per cent	80.16

Comparison of Ouachita coal tar with ordinary tar in fractionating^a.

Constituent	Ouachita tar, per cent	Ordinary tar, per cent
Water	11.3	15.0
Light oil	8.3	1.4
Middle oil	22.9	4.2
Heavy oil	12.6	24.0
Anthracene oil	40.4	.2
Pitch	4.5	55.0

^aCohen, Theoretical organic chemistry, p. 381.

MONITEAU COAL.

Destructive distillation of 25 lb. Moniteau coal.

Time, half hours	4	5	6	7	8	9	10	Totals
Temperature, degrees C.	400	540	600	700	760	775	785	
Oil, first condenser, c. c.	280 c. c. taken as d	160	210	60	15	30	30	905
Water, first condenser, c. c.	1.0842 for drain.	100	180
Oil, first condenser, sp. gr.	
Oil, second condenser, c. c.	1	2	1.027	1.049	340
Water, second condenser, c. c.	310	
Oil, second condenser, sp. gr.	
Oil, third condenser, c. c.	
Oil, third condenser, sp. gr.	280
Gas, cubic feet	
A ton of coal was found to give, according to this run, 32.23 gal. of oil; 318 gal. of water; 1080 lb. coke; and 8400 cu. ft. of gas.									
		10	17	29	25	14	4	1	105

Analysis of a general sample of the gas.

N ₂	7.2
H ₂ S and SO ₂2
CO ₂8
Illuminants	10.7
O ₂	1.1
CO	1.3
CH ₄	35.7
H ₂	41.7
Total	98.7

The general sample gave a B. t. u. value of 810.

Analysis of the gas given off at the end of each half hour during the distillation of the coal.

Time, half hours	1	2	3	4	5	6	7	8
N ₂	4.1	4.1	6.1	5.4	5.7	6.7
H ₂ S and SO ₂	2.0	1.3	.5	.2	.1	.2
CO ₂	3.1	.7	1.52	1.5
Illuminants	15.7	17.8	5.9	8.7	5.2	.2
O ₂	1.2	.6	.4	.3	.7	3.4
CO8	1.2	.7	2.0	1.3	1.7
CH ₄	43.5	36.1	24.9	34.0	32.7	15.8
H ₂	30.6	39.5	60.5	49.0	56.0	71.6
Totals	101.0	101.3	100.5	99.6	101.9	101.1

Analysis of the fractions from the fractionation of the general oils.

Temperature, degrees C.	70-125	125-170	170-200	200-210	210-235	235-270	270-305	305-320	320-355	355-400
Specific gravity8103	.8203	.8538	.9007	.9007	.9368	.9598	.9662	.9705	.9652
Fixed carbon, per cent00	.20	.20	.26	.28	.28	.45	1.53	1.63	---
Volatile matter, per cent	100.00	99.80	99.80	99.74	99.74	99.72	99.55	98.47	98.34	---
Ash, per cent	---	---	---	---	---	---	---	---	---	---
Limpid point	below-10° C. for the first five fractions	---	---	---	---	---	---	---	---	---
Flashing point	80° F. for (70-170)	160° F. for (170-235)	240° F. 260° F.	---	---	---	---	7° C.	23° C.	---
Burning point	120° F. for (70-170)	205° F. for (170-235)	335° F. 350° F.	---	---	---	---	330° F. for (305-400)	350° F. for (305-400)	---
Tar acid, per cent	1.99 for (70-170)	11.5 for (170-235)	10.40	---	---	---	---	9.00 for (305-355)	4.50	---
Tar base, per cent	0.00 for (70-170)	.8 for (170-235)	1.10	---	---	---	---	1.80 for (305-355)	22.00	---
Viscosity at 60 degrees C.	1.015 for (70-170)	1.11 for (170-235)	1.18	---	---	---	---	1.48 for (305-355)	---	---
Per cent of amount	5.52	3.06	5.30	6.28 for (200-235)	5.53	---	---	4.52	9.23	4.41
Bromine number	34.66	---	35.25	35.01 for (200-235)	34.88	---	---	---	---	---
Unsulphonated residue, per cent	---	---	---	---	---	---	---	9.00	---	---

Analysis of a general sample of the oil.

Specific gravity	1.0029
Water, per cent	12.76
Flashing point, degrees F.	90
Burning point, degrees F.	155
Viscosity, 60 degrees C.	1.92
Tar acid, per cent	16.45
Tar base, per cent	2.00
Volatile matter, per cent	91.75
Ash, per cent03
Fixed carbon, per cent	8.22
Free carbon, per cent20
Napthalene	0 00
Limpid point, degrees C.	2
Unsulphonated residue, per cent	9.06

Comparison of Moniteau coal tar with ordinary tar in fractionating^a

Constituent	Moniteau tar, per cent	Ordinary tar, per cent
Water	12.76	15.00
Light oil	8.59	1.40
Middle oil	11.58	4.20
Heavy oil	5.52	24.00
Anthracene	22.76	.20
Pitch	38.80	55.00

^aCohen, Theoretical organic chemistry, p. 381.

STOVER COAL.

Destructive distillation of 25 lb. of Stover coal.

Time, half hours	1	2	3	4	5	6	7	8	9	Totals
Temperature, degrees C.	220	400	435	530	600	630	650	675	760	
Oil, first condenser, c. c.	240	870	830	710	70	20	10	2750
Water, first condenser, c. c.	65	450	80	40	40	40	715
Oil, first condenser, sp. gr.	1.025	1.040	1.049	1.067	1.085	
Oil, second condenser, c. c.	80	50	20	150
Water, second condenser, c. c.	20	40	60
Oil, second condenser, sp. gr.	1.040	1.090	
Oil, third condenser, c. c.
Water, third condenser, c. c.
Gas, cubic feet	1	4	19	22	15	11	5	2	89

A ton of coal was found to give, according to this run, 64.88 gal. of oil; 16.38 gal. of water; 1160 lb. of coke; and 7120 cu. ft. of gas.

Destructive distillation of 25 lb. of coal.

Time, half hours	1	2	3	4	5	6	7	8	9	10	Totals
Temperature, degrees C.	260	375	405	515	580	650	715	740	750	
Oil, first condenser, c. c.	40	450	650	810	200	165	100	90	20	2525
Water, first condenser, c. c.	50	350	180	90	80	45	30	825
Oil, second condenser, c. c.	40	65	25	10	10	150
Water, second condenser, c. c.	5	5
Oil, third condenser, c. c.	170 c. c. taken as a drain at end of run	170
Gas, cubic feet	2	9	10	26	24	7	6	3	1	89

The specific gravity for the first, second and third condensers was the same as that given for the first run.

A ton of coal was found to give, according to this run, 60.13 gal. of oil; 17.54 gal. of water; 1180 lb. of coke; and 7060 cu. ft. of gas.

Analysis of a general sample of the gas.		Analysis of the gas given off at the end of each half hour during the distillation of the coal.									
N ₂	5.2	Time, half hours	1	2	3	4	5	6	7	8	
H ₂ S and SO ₂	4.5	N ₂	75.1	8.2	5.1	3.5	3.0	6.1	7.4	
CO ₂	3.0	H ₂ S and SO ₂	6.0	8.9	7.3	2.4	1.5	.5	
Illuminants.	3.9	CO ₂	5.0	3.6	2.7	1.6	.8	
O ₂4	Illuminants.	7.8	3.4	4.0	3.5	1.2	1.0	
CO	2.0	O ₂	2.6	.7	.5	.3	.7	.6	
CH ₄	34.2	CO	2.9	1.4	1.0	1.7	3.0	.9	
H ₂	48.2	CH ₄	5.2	39.2	44.3	28.1	28.0	23.3	
		H ₂	7.2	38.7	37.4	59.0	59.2	65.0	
Total.....	101.3	Totals.....	101.0	100.2	101.0	100.7	99.6	100.5	98.7		

The general sample gave a B. t. u. value of 781.

Analysis of the fractions from the fractionation of the general oils.

Temperature, degrees C. . .	70-125	125-170	170-200	200-210	210-235	235-270	270-305	305-320	320-355	355-400
Specific gravity8256	.8731	.9055	.9359	.9648	.9897	1.0199	1.0260	1.0460	1.0842
Fixed carbon, per cent03	.04	.05	.11	.21	.22	.22	.23	.25	.35
Volatile matter, per cent . .	99.97	99.95	99.94	99.87	99.77	99.76	99.76	99.75	99.73	99.63
Ash, per cent00	.01	.01	.02	.02	.02	.02	.02	.02	.02
Limpid point	below—10°C.	for first four fractions			—9°C.	—2°C.	3°C.	8°C.	16°C.	46°C.
Flashing point	104°F. for (70-170)	120°F. for (170-210)			140°F.	160°F.	200°F.	215°F.	235°F.	285°F.
Burning point	125°F. for (70-170)	230°F. for (170-210)			280°F.	330°F.	350°F.	360°F.	380°F.	-----
Tar acid, per cent	11.4 for (70-170)	30.0 for (170-210)			27.80	27.50	27.50	27.50	28.00	23.00
Tar base, per cent	1.4 for (70-170)	.5 for (170-210)			1.2	1.0	1.2	2.4	2.5	2.0
Viscosity at 60 degrees C..	1.07 for (70-170)	1.14 for (170-210)			1.18	1.30	1.48	2.19	2.35	5.30
Per cent of amount	1.16	3.16	4.36	2.49	6.04	7.51	5.71	4.36	9.07	17.18
Bromine number	96.35	96.36	102.60	102.80	106.70	97.80	-----	-----	-----	-----
Unsulphonated residue, % ..	33.70	40.00	50.00	31.58	21.62	23.33	26.66	21.00	21.00	28.52

Analysis of a general sample of the oil.

Specific gravity	1.063
Water, per cent	26.00
Flashing point, degrees F.	195
Burning point, degrees F.	
Viscosity, 60 degrees C.	3.25
Tar acid, per cent	32.00
Tar base, per cent50
Volatile matter, per cent	98.48
Ash, per cent03
Fixed carbon, per cent	1.49
Free carbon, per cent41
Napthalene, per cent	0.00
Limpid point, degrees C.	21
Unsulphonated residue, per cent	24.58

Comparison of Stover coal tar with ordinary tar in fractionating^a.

Constituent	Stover tar, per cent	Ordinary tar, per cent
Water	26.00	15.00
Light oil	4.32	1.40
Middle oil	12.89	4.20
Heavy oil	7.51	24.00
Anthracene oil	36.32	.20
Pitch	12.96	55.00

^aCohen, Theoretical organic chemistry, p. 381.

MARYVILLE COAL.

Destructive distillation of 35 lb. of Maryville coal.

	1	2	3	4	5	6	7	8	9	10	Totals
Time, half hours.....	1	2	3	4	5	6	7	8	9	10	
Temperature, degrees C.	---	---	390	440	570	650	740	840	850	850	
Oil, first condenser, c. c.	---	---	40	150	210	180	80	40	10	5	715
Water, first condenser, c. c.	---	---	450	620	500	270	170	80	40	10	2140
Oil, first condenser, sp. gr.	---	---	1.023	1.049	1.054	1.059	1.064	1.067	1.076	---	
Oil, second condenser, c. c.	---	---	15	---	---	40	20	---	---	---	75
Water, second condenser, c. c. ...	---	---	15	---	---	---	20	---	---	---	35
Oil, second condenser, sp. gr.	---	---	---	---	---	---	---	---	---	---	
Oil, third condenser, c. c.	---	---	---	---	---	---	---	---	---	---	
Oil, third condenser, sp. gr.	---	---	---	---	---	---	---	---	---	---	
Gas, cubic feet	---	---	5	11	16	19	37	38	16	7	149
A ton of coal was found to give, according to this run, 16.91 gal. of oil; 32.83 gal. of water; 1200 lb. of coke; and 8508 cu. ft. of gas.											

Destructive distillation of 25 lb. of Maryville coal.

	1	2	3	4	5	6	7	8	9	10	Totals
Time, half hours.....	1	2	3	4	5	6	7	8	9	10	
Temperature, degrees C.	---	450	535	635	733	856	875	938	966	966	
Oil, first condenser, c. c.	10	174	200	250	175	100	40	10	---	---	969
Water, first condenser, c. c.	200	535	380	300	125	50	---	---	---	---	1590
Oil, second condenser, c. c.	---	---	---	---	---	10	---	---	---	---	10
Water, second condenser, c. c.	---	---	---	---	---	---	---	---	---	---	
Oil, third condenser, c. c.	---	---	---	---	---	---	---	---	---	---	
Oil, third condenser, sp. gr.	---	---	---	---	---	---	---	---	---	---	
Gas, cubic feet	2	4	11	14	16	29	18	11	10	---	115
A ton of coal was found to give, according to this run, 23.88 gal. of oil; 33.60 gal. of water; 1160 lb. of coke; and 9200 cu. ft. of gas.											

The specific gravity for the first, second and third condensers was the same as that given for the first run.

A ton of coal was found to give, according to this run, 23.88 gal. of oil; 33.60 gal. of water; 1160 lb. of coke; and

9200 cu. ft. of gas.

Analysis of a general sample of the gas

N ₂	10.4
H ₂ S and SO ₂	1.1
CO ₂	1.4
Illuminants	3.6
O ₂	2.4
CO	8.0
CH ₄	29.4
H ₂	44.5
Total.....	100.8

The general sample gave a B. t. u. value of 592.

Analysis of the gas given off at the end of each half hour during the distillation of the coal.

Time, half hours .	1	2	3	4	5	6	7	8	9	10
N ₂ ^a	---	---	---	7.0	4.2	2.1	2.4	2.6	---	4.2
H ₂ S and SO ₂ ..	---	---	---	4.5	3.0	1.5	.7	.3	---	---
CO ₂	---	---	---	4.5	3.2	3.3	1.6	1.8	---	---
Illuminants ..	---	---	---	6.0	6.5	5.2	2.0	1.0	---	.2
O ₂	---	---	---	1.0	1.7	1.5	.2	.3	---	---
CO	---	---	---	7.3	7.0	6.7	10.5	12.1	---	5.0
CH ₄	---	---	---	53.2	39.2	25.0	9.5	5.6	---	6.1
H ₂	---	---	---	16.5	35.2	54.7	73.1	76.3	---	84.5
Totals.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

^aNitrogen obtained by difference.

Analysis of the fractions from the fractionation of the general oils.

Temperature, degrees C.	70-170	170-200	200-210	210-235	235-270	270-305	305-320	320-355	355-400
Specific gravity8878	.9522	.9681	.9919	1.0170	1.0371	1.0411	1.0695	1.1041
Fixed carbon, per cent03	.44	.47	.50	.60	.73	.83	1.02	2.28
Volatile matter, per cent	99.97	99.56	99.42	99.49	99.38	99.25	99.15	98.98	97.70
Ash, per cent00	.00	.00	.01	.02	.02	.02	.02	.02
Limpid point	below -10°C.	for the first five fractions							
Flashing point	90°F.	125°F.	180°F.	220°F.	250°F.	320°F.	4°C.	22.5°C.	49.0°C.
Burning point	100°F.	170°F.	270°F.	310°F.	340°F.				
Tar acid, per cent	8.00	31.72	21.90	21.43	21.30	20.10	17.50	17.50	36.54
Tar base, per cent50	.50	.50	1.40	1.50	1.00	1.00	1.00	3.00
Viscosity at 60 degrees C.	1.013	1.073	1.150	1.180	1.250	1.488	2.100	2.800	3.700
Per cent of amount	4.90	7.50	3.12	10.00	11.06	8.66	5.33	9.33	16.00
Bromine number	22.72	24.92	26.50	26.50	27.71	28.49	28.60	---	---
Unsulphonated residue, per cent .	21.00	16.20	13.30	7.15	6.90	6.00	---	5.71	3.44

Analysis of general sample of oil.

Specific gravity	1.066
Water, per cent	7.50
Flashing point	
Burning point	
Viscosity at 60 degrees C.	
Tar acid, per cent	42.50
Tar base, per cent	2.00
Volatile matter, per cent	89.13
Ash, per cent04
Fixed carbon, per cent	10.83
Free carbon, per cent	5.08
Napthalene, per cent	0.00
Limpid point, degrees C.	1
Unsulphonated residue, per cent.....	16.66(?)

Comparison of Marysville coal tar with ordinary tar in fractionating^a.

Constituent	Maryville tar, per cent	Ordinary tar, per cent
Water	7.50	15.00
Light oil	4.90	1.40
Middle oil	20.62	4.20
Heavy oil	11.06	24.00
Anthracene oil	39.32	.20
Pitch	16.60	55.00

^aCohen, Theoretical organic chemistry, p. 381.

WYOMING COAL.

Analysis of a general sample of the gas.

N ₂	9.1
H ₂ S and SO ₂	0.1
CO ₂	16.4
Illuminants	1.5
O ₂	1.0
CO	11.2
CH ₄	22.4
H ₂	38.5
<hr/>	
Total	100.2

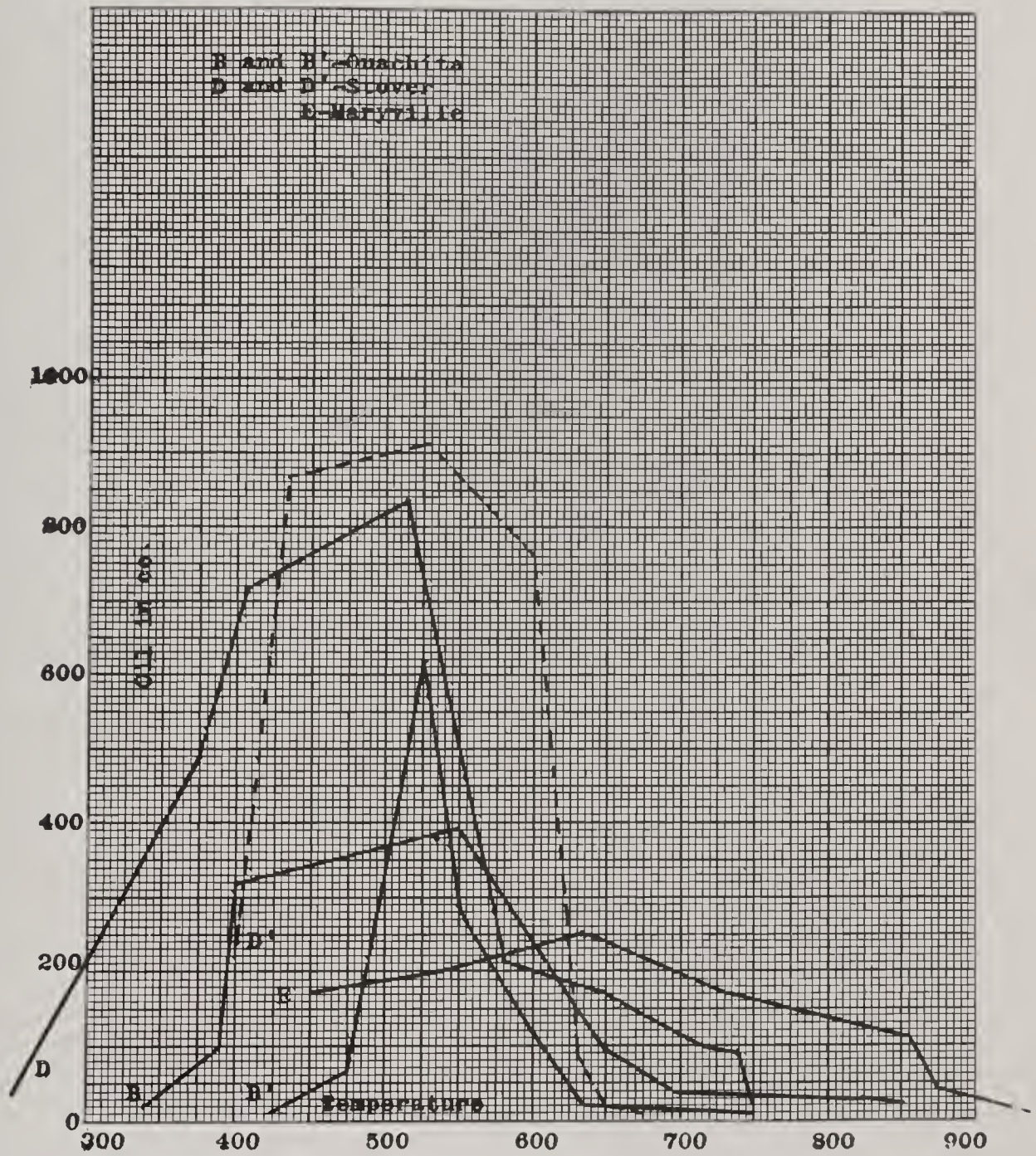
Analysis of the gas given off at the end of each half hour during the destructive distillation of the coal.

Time, half hours	1	2	3	4	5	6
N ₂	-----	7.4	6.5	9.4	13.3	15.0
H ₂ S and SO ₂	-----	-----	-----	-----	-----	1.0
CO ₂	-----	24.0	17.0	7.4	6.5	3.0
Illuminants	-----	1.5	1.9	2.0	2.6	2.0
O ₂	-----	1.0	1.3	.9	.9	.5
CO	-----	10.0	8.0	9.9	6.8	11.5
CH ₄	-----	22.4	22.2	25.7	29.0	20.7
H ₂	-----	34.0	44.5	43.5	41.5	47.0
<hr/>		<hr/>				
Totals		100.3	101.4	98.8	100.6	100.7

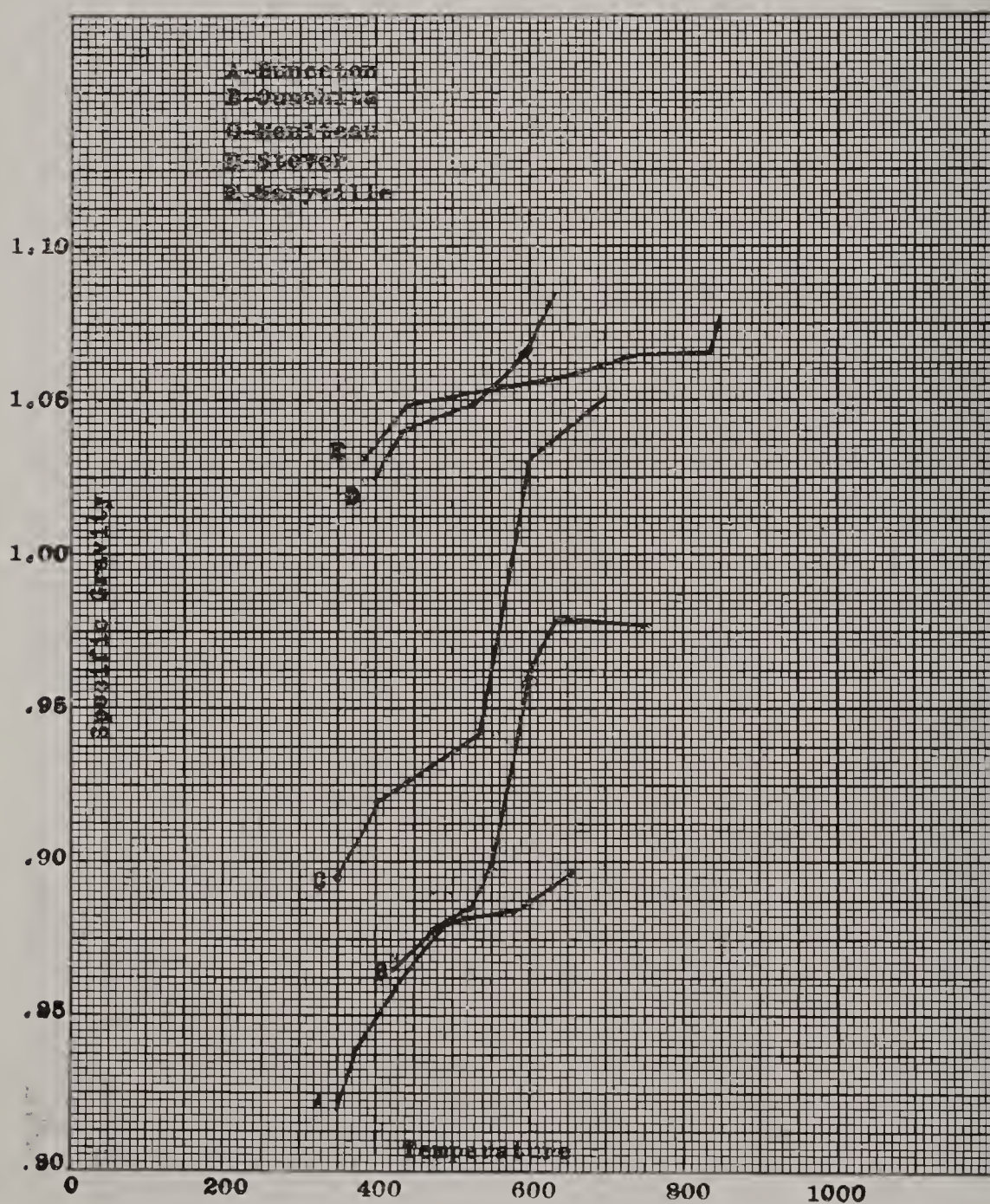
Analysis of a general sample of the oil.

Specific gravity	1.009
Water, per cent	51.00
Flashing point, degrees F.	160
Burning point, degrees F.	250
Viscosity, 60 degrees C.	2.206
Tar acids, per cent	26.00
Tar bases, per cent60
Volatile matter, per cent	96.32
Ash, per cent	0.00
Fixed carbon, per cent	3.68
Free carbon, per cent	2.74
Napthalene, per cent	0.00
Limpid point, degrees C.	1
Unsulphonated residue, per cent	36.66

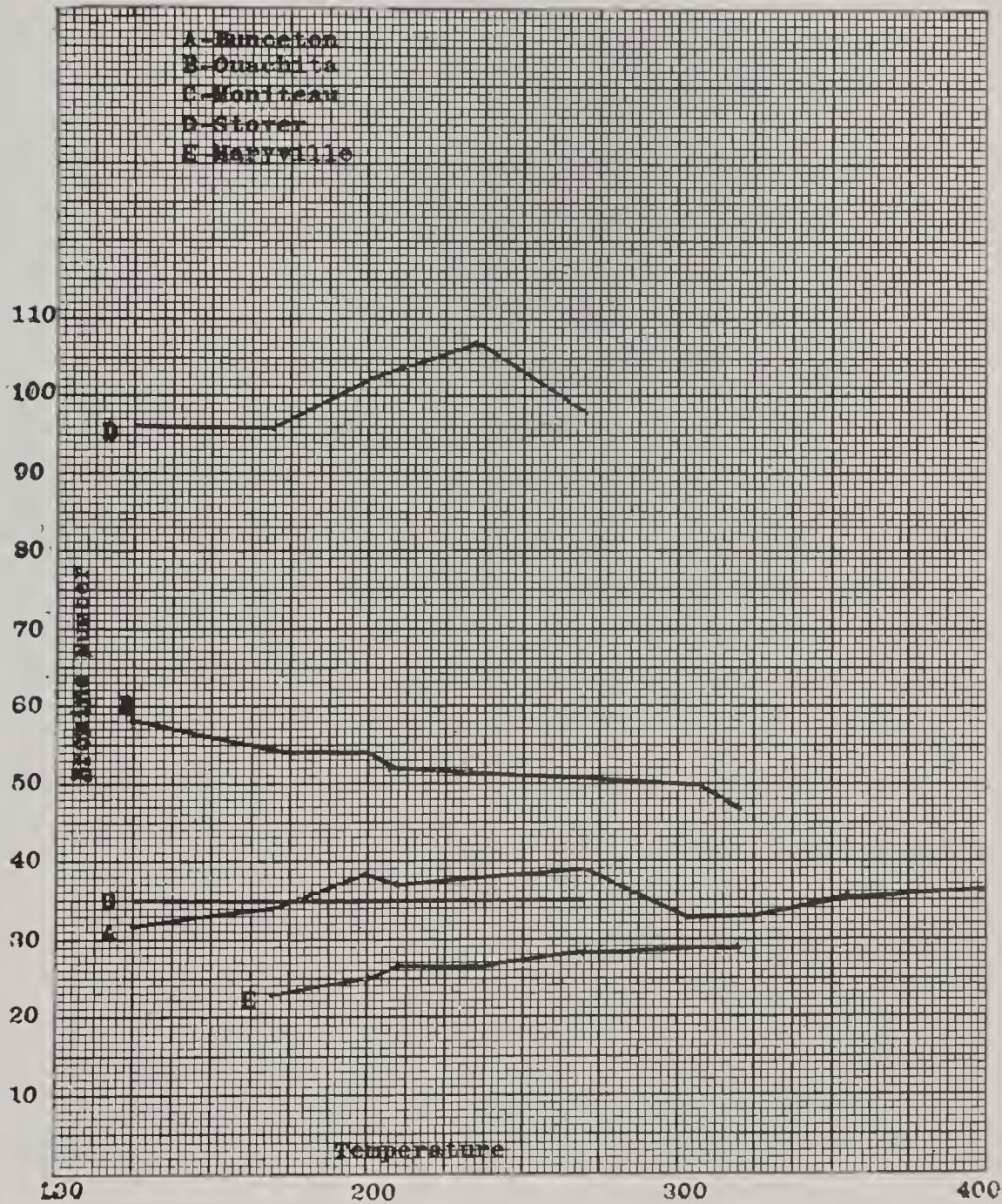
A ton of coal was found to give, as calculated from a run, 2.59 gal. of oil; 127 gal. of water; 900 lb. of coke; and 8183 cu. ft. of gas.



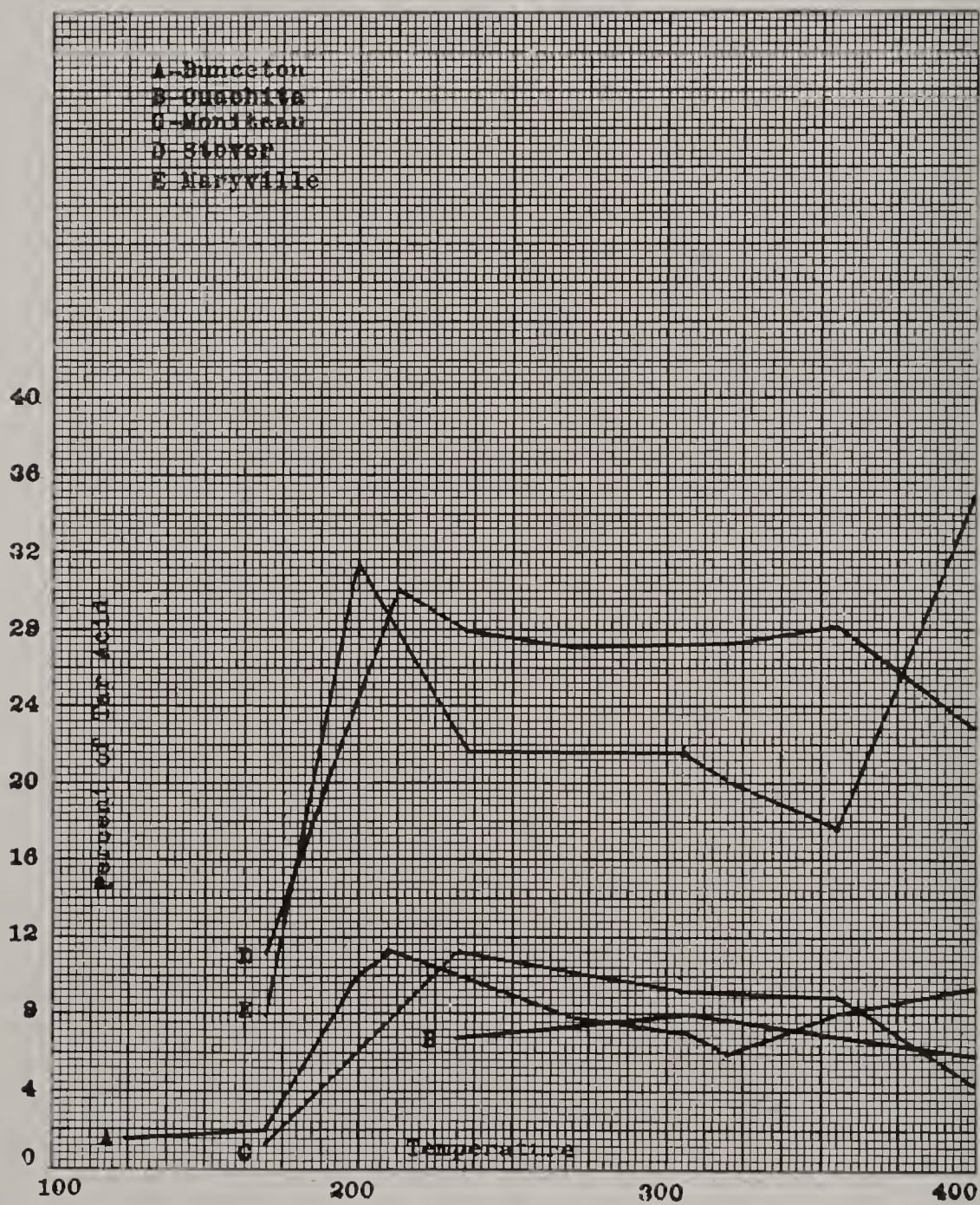
OIL PRODUCTION AND TEMPERATURE.



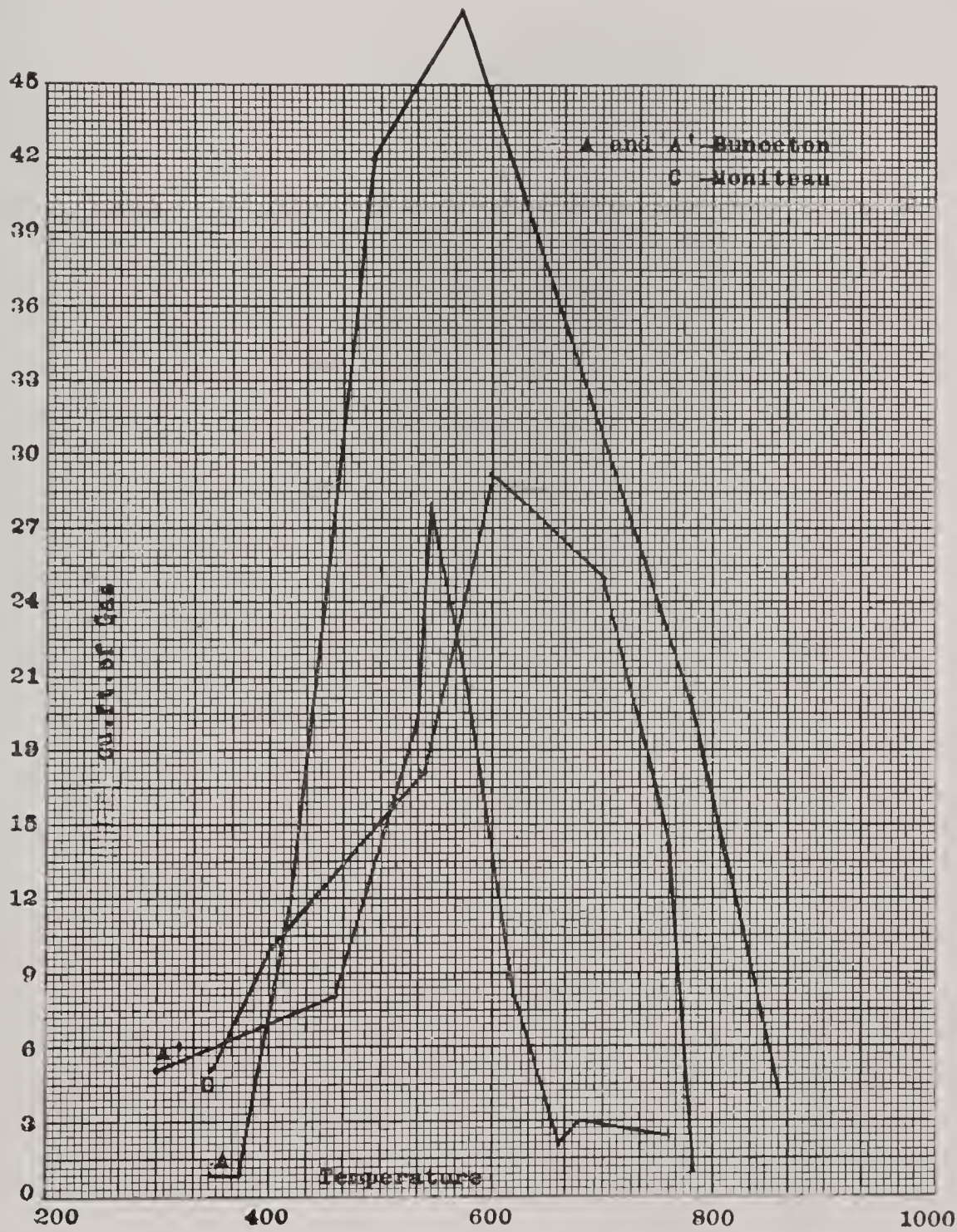
SPECIFIC GRAVITY OF OILS AND DISTILLATION FRACTIONS.



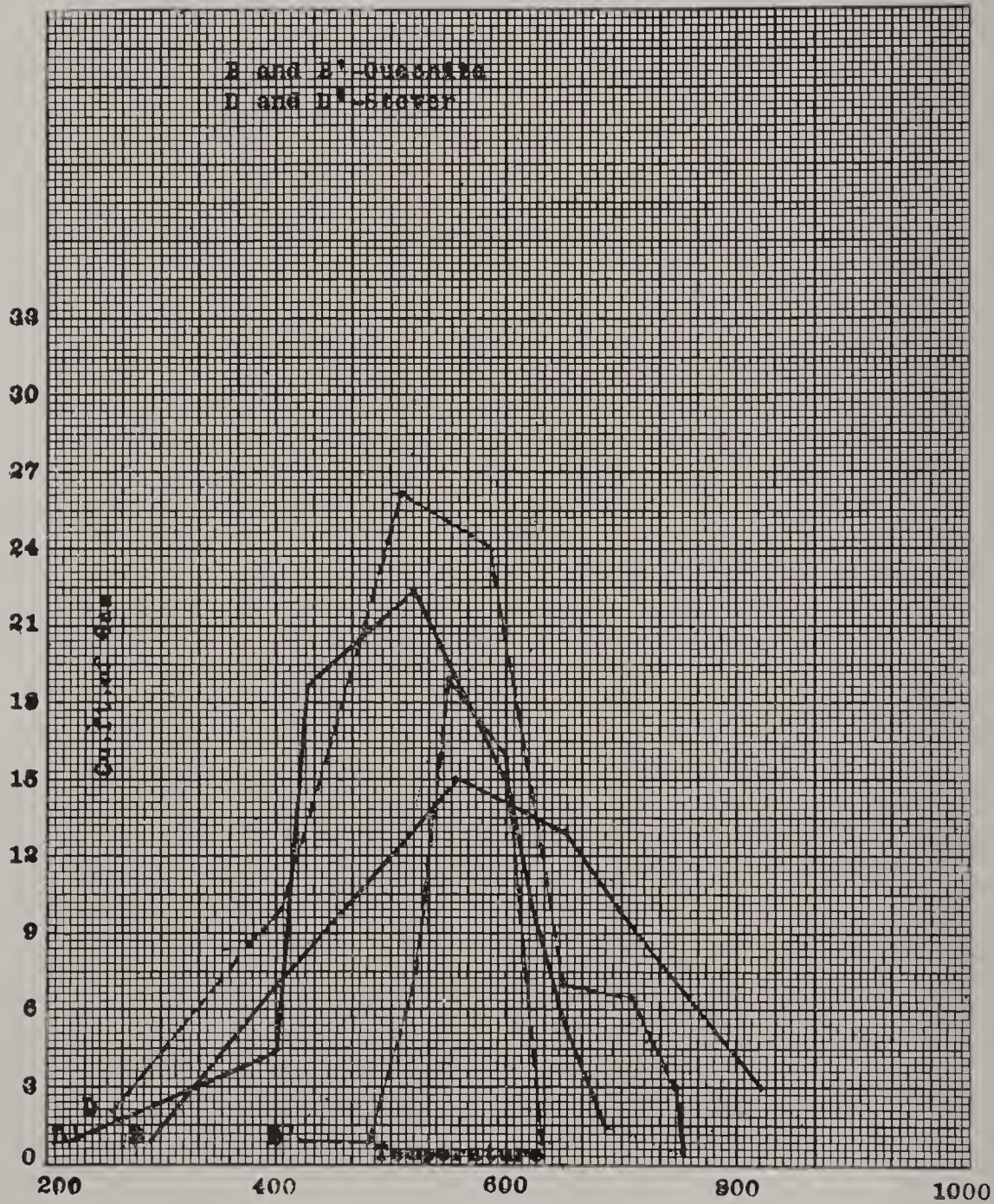
BROMINE NUMBER AND TEMPERATURE OF DISTILLATION.



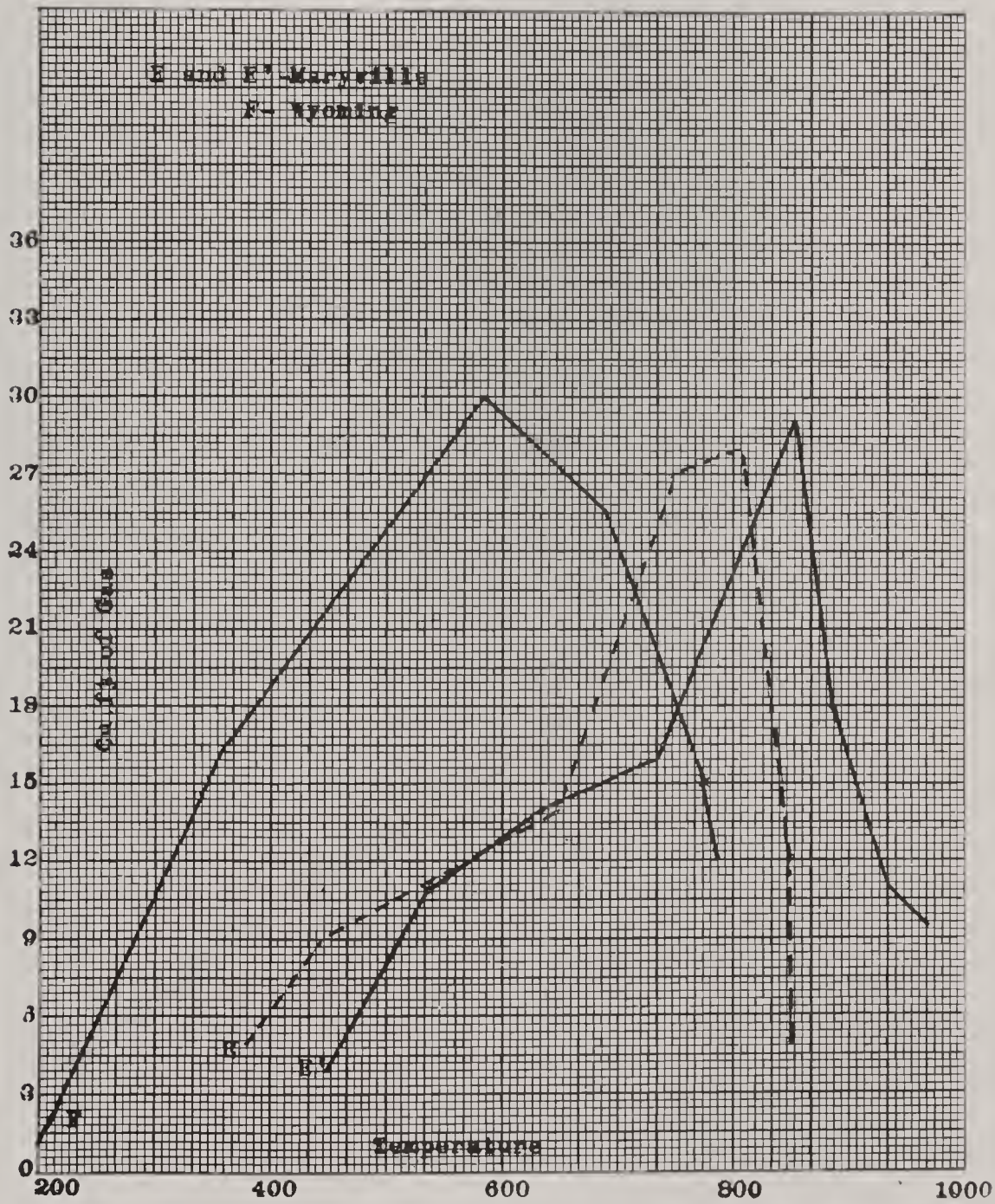
TAR ACIDS AND TEMPERATURE OF DISTILLATION.



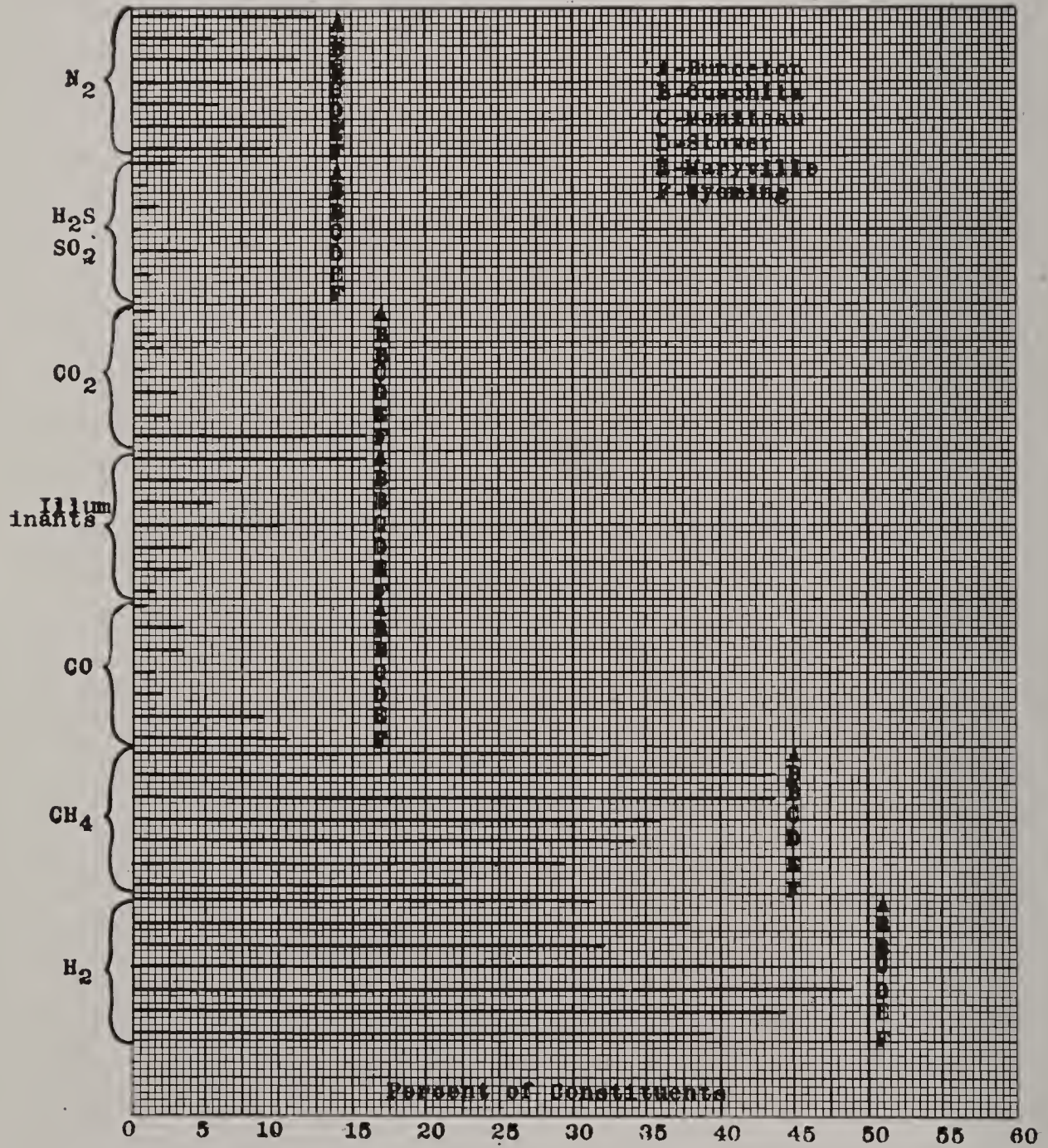
GAS PRODUCTION AND TEMPERATURE.
Bunceton and Moniteau Coals.



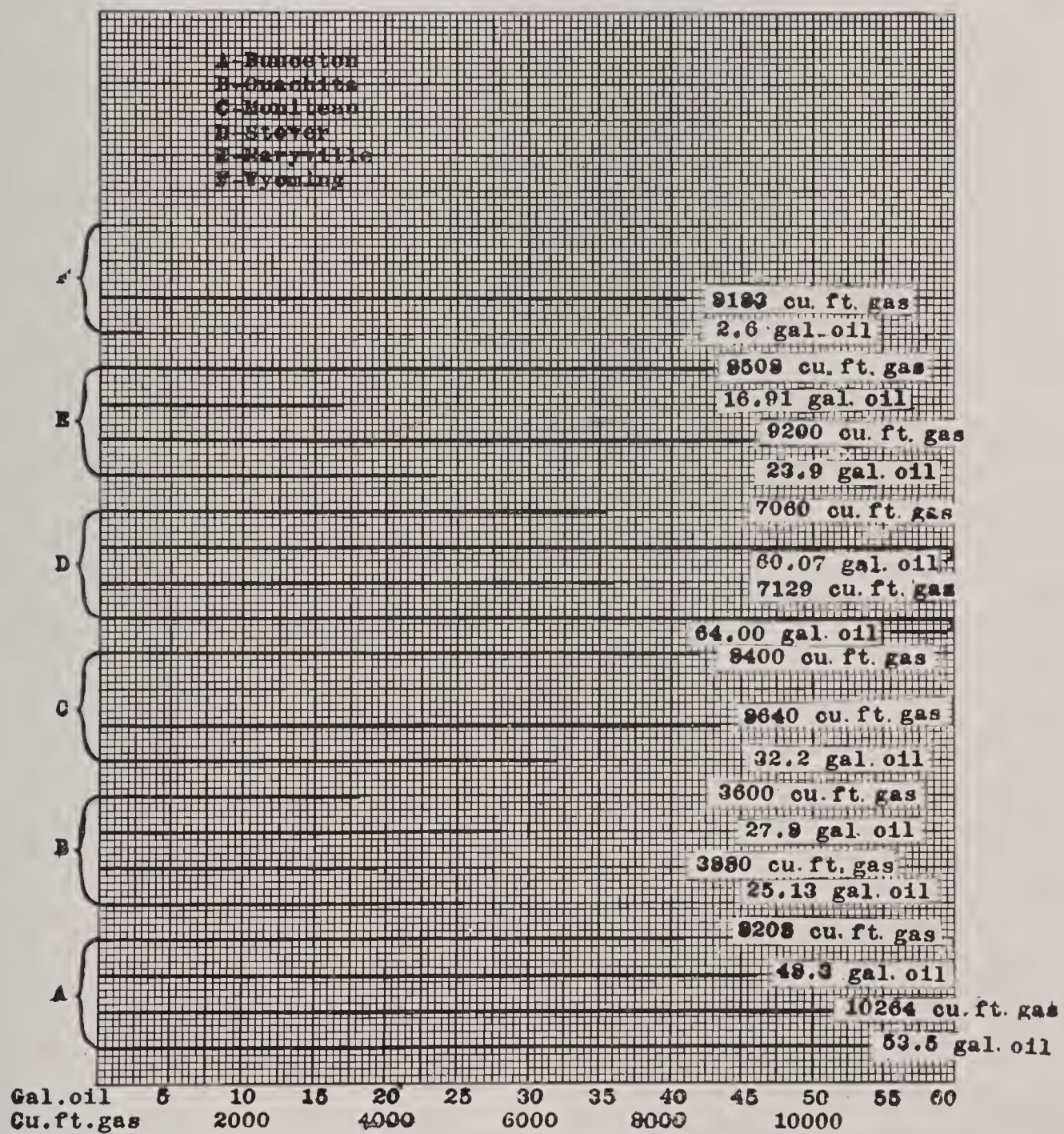
GAS PRODUCTION AND TEMPERATURE.
Ouachita and Stover Coals.



GAS PRODUCTION AND TEMPERATURE.
Maryville and Wyoming Coals.



CONSTITUENTS OF GASES, PER CENT.



OIL AND GAS PRODUCTION, PER TON.

Discussion of Oil Production With Time of Heating and Temperature.

In the first stages of heating, the vaporization of the hygroscopic moisture undoubtedly carries over into the condensers some of the hydrocarbons. Consequently, a wide range in the quantity of water in different coals makes impossible a very close comparison of the amounts of oils secured at low temperature distillations. In the first stages of heating the hygroscopic water disappears and this can be considered complete at about 200 degrees C¹⁰. Above this temperature the products of the decomposition depend upon the constituents of the coal¹¹: cellulosic matter yields oil, water and gases; resinous constituents decomposing at a higher temperature, yield less water and gases and more paraffin, olefines, olefiant gases and hydrogen.

According to Burgess and Wheeler¹² all coals have a well defined decomposition point between 700 and 800 degrees C., which corresponds to a marked increase in quantity of hydrogen evolved. But with the cannel coals, the data would justify saying that the maximum decomposition temperature was between 600 and 700 degrees, if the maximum production of oil, water and hydrogen are considered. This can be seen from the curves 3 and 4, when the average temperature of the interior and exterior of the retort is considered. The greatest yield of water was obtained by slow heating and the greatest amount of oil with fast heating¹³. It will be noted that the maximum oil yields for the different runs do not show a very wide variation for the same coal.

The path of the gas from the retort to the condensers has a great influence upon the composition of the oils¹⁴. When the vapors are subjected to high temperatures, the hydrocarbons are cracked, giving an oil of higher specific gravity and more free carbon. The increase of specific gravity of the oils driven off with different stages in the process of distillation is due, in a measure, to the cracking and condensation of some of the hydrocarbons at the higher temperatures. The percentages of free carbon in these successive periods of heating increase as the temperature of the retort rises.

¹⁰Wilson, E. B., Adaptability of West Virginia coals to by-product coking: *Colliery Eng.*, vol. 35, pp. 151-153, 1914.

¹¹Taylor, G. B., and Porter, H. C., The primary volatile products of the carbonization of coal: U. S. Bureau of Mines, Tech. Paper No. 140, p. 51, 1916.

¹²Burgess, M. J., and Wheeler, R. V., The volatile constituents of coal: *Jour. Chem. Soc.*, vol. 97, pp. 1917-1935, 1910.

¹³Rau, O., and Lambris, G., Die Wasserbildung bei der trockenem Destillation der Brennstoffe: *Jour. f. Gasbel.*, Jahrg. 56, pp. 533-536, 557-564, 589-591, 1913.

¹⁴Perry, R. P., Tar distillation in United States: Eighth International Congress of Applied Chemistry; vol. 10, p. 233, 1912.

Parr and Olin^{15 16} coked some Illinois coals by the use of superheated steam at 400 to 500 degrees C., in which only 18 to 20 per cent of the volatile matter was left behind in the coke; but the gas produced was only 10 per cent of that compared with high temperature distillation. The tar produced contained only 30 per cent pitch with little free carbon. It had a lower specific gravity and a higher per cent of tar acids.

Cannel coals at the lower temperature distillation gave a light oil, having a minimum of tar acids for the coal. As to whether these lighter oils contain a very high per cent of olefines, will depend upon the coal and the manner of distillation. Undoubtedly the bromine number of the cuts would show the same general relation to the oils obtained by successive stages of carbonization. The curves in Fig. 6 show that there is no regularity, the bromine number in some cases showing an increase and in other a decrease. The unsulphonated residues seem to pass through a maximum at about 200 degrees C., in the fractionation. This perhaps is in the same general order as would be found in the oils coming from the retorts in the process of coking.

Production of gases at successive temperatures of distillation.

Here again it is found that the maximum evolution of gases is at a lower temperature than with bituminous coals. However, it is impossible from analysis to differentiate entirely between time and temperature. The coal in contact with the hotter outside of the retort will yield more of its gas than that in the interior where the temperature is not so high, and it may be some time, comparatively, before it can reach the same temperature.

In the first stages when the temperature is below the decomposition point of the "cellulosic matter," paraffin hydrocarbons predominate and a small per cent of hydrogen is produced. The sudden rise in percentage of hydrogen is seen to be at a lower temperature than is found to be the case with bituminous coals.

The evolution of hydrogen sulphide and sulphur dioxide proceeds in the same manner as in other coals, gradually decreasing as the sulphur content diminishes. The sulphur compounds in cannel coal probably do not differ from those found in bituminous coals. It must be borne in mind that the percentage present in the gases for the different stages of carbonization of the coal is different from the actual quantity evolved. The quantity for most of the gases is the greatest at the "critical

¹⁵Parr, S. W., and Olin, H. L., The coking of coals at low temperatures: Univ. of Illinois, Eng. Exper. Sta., Bull. 60, 1912.

¹⁶Parr, S. W., and Olin, H. L., The coking of coals at low temperatures, with special reference to the properties and composition of the products: Univ. of Illinois, Eng. Exper. Sta., Bull. 78, 1915.

point" in the process of carbonization. In the tables the percentage composition of different constituents is given.

As is to be expected, carbon dioxide decreases with the decomposition of the oxygenated constituents, while the carbon monoxide does not show such variation, as a whole, for the different runs. Burgess and Wheeler¹⁷ explain this fact as due to the elimination of water from the hydroxy compounds and the subsequent reaction of the steam thus formed with the carbon.

The gas has a very disagreeable odor due to the sulphur compounds, and when burned in the laboratory the sulphur dioxide formed soon contaminated the air so as to make it unbearable. For domestic use these sulphur compounds would have to be removed. No attempt was made to remove the hydrogen sulphide as is done in commercial plants, but no doubt this could be accomplished by the same means.

The B. t. u. of the gases of the cannel coals runs high, this making the gas valuable for heating purposes.

As for the benzene, toluene and xylenes¹⁸, less was found than in the bituminous coal examined. The cannel coals did not give sufficient amounts of benzene, toluene and xylene to secure quantitative tests. With the view of determining these products the authors would suggest that these coals be carbonized rapidly at a higher temperature than they used so as to crack some of the light oils. The coking temperature for a maximum yield of benzene and toluene requires only a moderate heat, and from the fact that cannel coals are richer in the lighter oils, it seems that it might be possible to work these coals for these products

Summary.

1. Five different cannel coals were subjected to destructive distillation in a gas-fired horizontal retort and compared with a bituminous coal coked under similar conditions.

2. The decomposition temperature of cannel coal is much lower than that of bituminous coals.

3. Different cannel coals show a wide variation in the yields of oils or tars. In general, cannel coals yield a much larger amount of oil than do bituminous coals.

4. The oils from cannel coals have a low specific gravity and consist chiefly of paraffin hydrocarbons. These oils resemble the oils obtained by low-temperature carbonization of bituminous coals and they can easily be explained as due to the fact that cannel coals have a lower decomposition temperature. This low decomposition temperature is due to a large amount of cellulosic matter in the coal.

¹⁷Burgess, M. J., and Wheeler, R. V., Volatile constituents of coal: Jour. Chem. Soc., vol. 99, pp. 649-667, 1911.

¹⁸Sperr, F. W., Laboratory method for benzol-recovery plant operation: Met. & Chem. Eng., vol. 16, pp. 549, 586, 642, 1917.

5. Cannel coals yield a greater amount of gas than do bituminous coals, and this gas has a high calorific and illuminating value. Again, this is what is found in the coking of bituminous coals at a low temperature. With the removal of sulphur compounds, cannel coal gas would be valuable for illuminating uses.

6. The yield of ammonium sulphate per ton cannel coals is less than that from bituminous coals. Coking at a higher temperature would undoubtedly give more of this product.

7. There is a wide range in the cokability of cannel coals. Only two of the coals examined, Bunceton and Stover, gave a coke of commercial value.

8. The cannel coals examined would not be a source for toluene. The gases and oils are very low or possibly entirely devoid of benzene and naphthalene derivatives. This is due to the low temperature cracking of the compounds in the coal. Under suitable conditions, these oils, in all probability, could be cracked and used as a source for aromatic hydrocarbons, but the cost of such an operation would make it impossible to compete with the same products obtained by cracking the cheaper petroleum oils.

9. The amount of free carbon in the tars is low and the pitch content is low. There is a large amount of low distilling oils in the crude tar.

10. The oils obtained would have little commercial value as such, the only use for them would be as a fuel. A good cannel coal might be of great value to mix with bituminous coals for the making of gas, because of the high illuminating value of the cannel coal gas.

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